

## PATENT ABSTRACTS OF JAPAN

Citation 1'

(11)Publication number : 08-059994

(43)Date of publication of application : 05.03.1996

(51)Int.Cl.

C08L 83/04

A61K 7/00

C08J 3/03

C08J 3/075

C08K 5/00

(21)Application number : 07-120294

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(22)Date of filing : 19.05.1995

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(30)Priority

Priority number : 95 385293

Priority date : 10.02.1995

Priority country : US

94 321640

11.10.1994

94 250124

27.05.1994

US

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(54) PREPARATION OF MICROEMULSION OF AMINOSILICONE FLUID/MQ RESIN MIXTURE

(57)Abstract:

PURPOSE: To obtain the subject microemulsion being transparent or translucent and useful as a personal care product by adding a surfactant having a high phase inversion temperature to a microemulsifiable silicone, adding water to the mixture at that temperature, and quickly adding water to the mixture after acidification.

CONSTITUTION: The objective emulsion is obtained by

adding (B) a surfactant having a phase inversion temperature in the range of about 45 to about 95° C

(desirably, about 55 to about 95° C) to (A) a

microemulsifiable silicone represented by formula I

[wherein R is a 1-6C hydrocarbon group; Q is the

formula: -R<sub>1</sub>HZ [wherein R<sub>1</sub> is H or a divalent

connecting group; Z is an amino-containing polar group

such as of formula II or NX<sub>2</sub> (wherein X is H or a 1-12C

alkyl; (z) is 1 or greater; and zz is 0 or greater); (a) is 0

to about 2; (b) and (c) are each about 1 to about 3; a+b≤

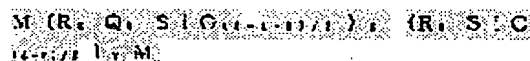
3; (x) is 5-2,000; (y) is 2-10,000; and M is an end stop

group of the silicone]], heating the resulting mixture to

about 45 to 95° C, adding water to the mixture, adding an acid (desirably, acetic acid or the

like), to it, and further adding water. It is desirable that the amino content of the silicone is

about 0.10 to about 10 meq/g.



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## LEGAL STATUS

[Date of request for examination] 10.05.2002

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

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## CLAIMS

## [Claim(s)]

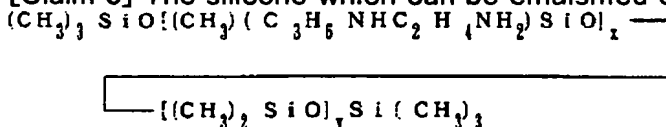
[Claim 1] (a) The translucent oil-water-type micro emulsion containing the silicone which can be emulsified detailed, and the (b) abbreviation 45 - surfactant [ which has the phase inversion temperature of the range of about 95 degrees C ], and (c) water.

[Claim 2] The silicone which can be emulsified detailed The inside of a formula  $M(R_a Q_b SiO_{(4-a-b)/2})_x y M(R_c SiO_{(4-c)/2})$  [formula, R is a hydrocarbon group which has the carbon atom of 1 - six abbreviation. Q The inside of a general formula  $-R_1 HZ$  [formula, R1 Whether it is the bivalent connection machine combined with hydrogen and Basis Z, and consists of a carbon atom and a hydrogen atom Or a carbon atom, It consists of a hydrogen atom and an oxygen atom, or consists of a carbon atom, a hydrogen atom, and a nitrogen atom, and Z is formula  $-N(CH_2)_z$   $zzNX (CH_2)_2$  and  $NX_2$  (as for X of  $X_2$ , each becomes independent among a formula). Although it is chosen from the groups which consist of hydrogen and the alkyl group of 1-12 carbon atoms, z is one or more and zz is zero or more When zz is 0, Z However, formula  $-NH(CH_2)_z NH_2$  It is the polar group which has} which is the amino content machine chosen from the groups which consist of the basis which it has. (however, the inside z of a formula is one) or more — having — a is the ranges from about 0 to about 2, and b is the ranges from about 1 to about 3. However, a+b is three or less and c is the number of the ranges from about 1 to about 3. x — five — from — about — 2,000 — up to — the range — a number — it is — y — about — 20 — from — about — 10,000 — up to — the range — a number — it is — M — silicone — an end — a halt — a machine — it is —] — having — silicone — it is — being according to claim 1 — micro — an emulsion .

[Claim 3] The micro emulsion according to claim 1 the range of whose phase inversion temperature of a surfactant is about 55 to about 95 degrees C.

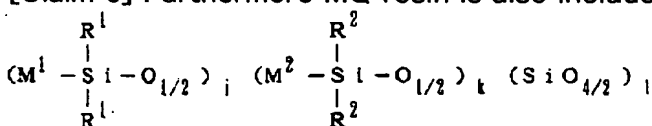
[Claim 4] The micro emulsion according to claim 2 whose amino content of silicone is the rang of about 0.10 - about 10 meq/g.

[Claim 5] The silicone which can be emulsified detailed is a formula further. [Formula 1]



It is a micro emulsion containing the silicone which has (x are the number of the ranges from about 5 to about 2,000 among a formula, and y is the number of the ranges from about 800 to about 10,000) according to claim 4.

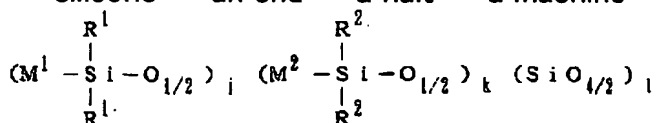
[Claim 6] Furthermore MQ resin is also included and this MQ resin is a formula. [Formula 2]



It is chosen among [formula from the groups of the alkyl hydrocarbon group in which R1 and R2 have indep ndently [ both ] 1-12 carbon atoms, and, as for M1 and M2, both become

independent. It is chosen from the groups which consist of the arbitrary combination of the alkyl hydrocarbon group which has a phenyl, a phenethyl, a polyether, hydrogen, and 1-12 carbon atoms. However, Subscripts j, k, and l are micro emulsions according to claim 2 whose viscosity of this MQ resin it is chosen from the groups of MQ resin which have] which fills following relation  $0.5 \leq (j+k) / l \leq 4.0$ , and is the range of 50 to 5,000 centistokes at 25 degrees C.

[Claim 7] (a) Inside of a (i) formula  $M(R_a Q_b SiO_{(4-a-b)/2})_x y M(R_c SiO_{(4-c)/2})$  [formula, R is a hydrocarbon group which has the carbon atom of 1 - six abbreviation. Q The inside of a general formula-R1 HZ{formula, R1 Whether it is the bivalent connection machine combined with hydrogen and Basis Z, and consists of a carbon atom and a hydrogen atom Or a carbon atom, It consists of a hydrogen atom and an oxygen atom, or consists of a carbon atom, a hydrogen atom, and a nitrogen atom, and Z is formula-N(CH<sub>2</sub>)<sub>z</sub> zzNX (CH<sub>2</sub>)<sub>2</sub> and NX<sub>2</sub> (as for X of X<sub>2</sub>, each becomes independent among a formula). Although it is chosen from the groups which consist of hydrogen and the alkyl group of 1-12 carbon atoms, z is one or more and zz is zero or more When zz is 0, Z However, formula-NH(CH<sub>2</sub>)<sub>z</sub> NH<sub>2</sub> It is the polar group which has} which is the amino content machine chosen from the groups which consist of the basis which it has. (however, the inside z of a formula is one) or more — having — a is the ranges from about 0 to about 2, and b is the ranges from about 1 to about 3. However, a+b is three or less and c is the number of the ranges from about 1 to about 3. x is the number of the ranges from 5 to about 2,000, and y is the number of the ranges from about 20 to about 10,000. The inside of the silicone which has] whose M is the end halt machine of silicone, and a (ii) formula  $M(R_a Q_b SiO_{(4-a-b)/2})_x y M(R_c SiO_{(4-c)/2})$  [formula, R is a hydrocarbon group which has the carbon atom of 1 - six abbreviation. Q The inside of a general formula-R1 HZ{formula, R1 Whether it is the bivalent connection machine combined with hydrogen and Basis Z, and consists of a carbon atom and a hydrogen atom Or a carbon atom, It consists of a hydrogen atom and an oxygen atom, or consists of a carbon atom, a hydrogen atom, and a nitrogen atom, and Z is formula-N(CH<sub>2</sub>)<sub>z</sub> zzNH (CH<sub>2</sub>) (although z is one or more among a formula and zz is zero or more). When zz is 0, Z However, formula-NH(CH<sub>2</sub>)<sub>z</sub> NH<sub>2</sub> It is the polar group which has} which is the amino content machine which it has. (however, the inside z of a formula is one) or more — having — a is the ranges from about 0 to about 2, and b is the ranges from about 1 to about 3. however — a+b — three — less than — it is — c — about — one — from — about — three — up to — the range — a number — it is — x — one — from — about — 20 — up to — the range — a number — it is — y — about — 20 — from — about — 800 — up to — the range — a number — it is — M — silicone — an end — a halt — a machine — it is —] — having — silicone — [Formula 3]



It is chosen among [formula from the groups of the alkyl hydrocarbon group in which R1 and R2 have independently [ both ] 1-12 carbon atoms, and, as for M1 and M2, both become independent. It is chosen from the groups which consist of the arbitrary combination of the alkyl hydrocarbon group which has a phenyl, a phenethyl, a polyether, hydrogen, and 1-23 carbon atoms. However, Subscripts j, k, and l fill following relation  $0.5 \leq (j+k) / l \leq 4.0$ . Moreover, the silicone which has] in which the MQ resin concerned has the viscosity of the range of 50 to 5,000 centistokes at 25 degrees C, The silicone which can be emulsified detailed is chosen from the groups which consist of such mixture. a row — (iv) — (b) The surfactant which has the phase inversion temperature of the range from about 45 degrees C to about 95 degrees C is added to the aforementioned silicone. (c) The manufacture method of a micro emulsion including heating the aforementioned silicone and the aforementioned surfactant to the temperature of the range from about 45 degrees C to about 95 degrees C, adding (d) water I, adding the (e) acid, and adding (f) water II.

[Claim 8] The method according to claim 7 of carrying out a process (d) and (e) simultaneously substantially.

[Claim 9] The method according to claim 7 of dissolving the acid of a process (e) in the water of

a process (d), considering as an acid solution, and adding this acid solution into the mixture of (a) and (b).

[Claim 10] The method according to claim 7 of choosing an acid from the groups which consist of an acetic acid, a hydrochloric acid, hypophosphorous acid, a lactic acid, a propionic acid, a glycolic acid, a formic acid, and a nitric acid.

[Claim 11] The personal care product containing a micro emulsion according to claim 1.

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## CLAIMS

## [Claim(s)]

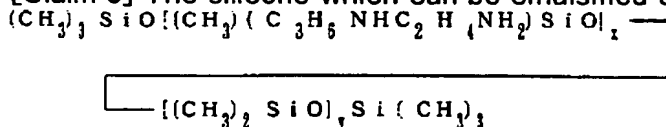
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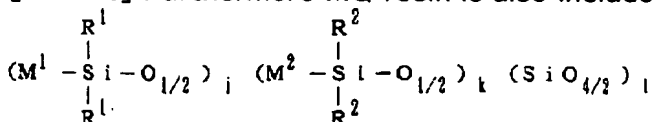
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## DETAILED DESCRIPTION

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### [Detailed Description of the Invention]

#### [0001]

[Industrial Application] this invention relates to the method of preparation of a micro emulsion. The method of this invention blends amino silicone fluid-MQ resin mixture with a surfactant with high phase inversion temperature, adds the water of almost the equivalence in temperature almost equal to the phase inversion temperature of this surfactant, and after it acidifies, it consists of adding water quickly. Furthermore, this invention relates also to the personal care product containing the micro emulsion prepared by the method of this invention.

#### [0002]

[Description of the Prior Art] It is related with the method of creating a micro emulsion blend average grain size has about 0.001 – 0.05 microns of abbreviation, and this invention contains a resin or MQ resin, and a surfactant with high phase inversion temperature, including the low silicone of an amino content at least one sort. Furthermore, this invention relates also to the personal care product containing this micro emulsion.

[0003] Various personal care products understand that the micro emulsion containing a silicone fluid is useful. A mean particle diameter usually makes preferably the 0.040 microns or less of the 0.05 microns or less of the terms "the micro emulsion (liquid which carried out detailed emulsification)" used on these specifications the thing containing the small drop which is 0.025 microns or less for a diameter which points out and says a stable system transparently, mechanically, and thermally with a diameter for a diameter. Since the size of a drop is small, an emulsion is highly transparent.

[0004] Use of a micro emulsion is well-known in the industry. For example, U.S. Pat. No. 4,797,272 and GI (Gee) of Lynn and others (Linn) Please refer to 4,620,878. The water-type micro emulsion constituent in an oil which is the range whose average drop sizes are about 0.001 – 0.200 microns of abbreviation is indicated by U.S. Pat. No. 4,797,272 of Lynn and others (Linn). GI (Gee) The polyorganosiloxane emulsion containing the polyorganosiloxane which contains at least one piece or at least one silanol group for the amino group combined with the silicon of a siloxane by Si-C combination or Si-O-C combination or a polar basis like an ammonium in U.S. Pat. No. 4,620,878, and at least one sort of surfactants insoluble to this polyorganosiloxane is indicated. If water is added, a translucent concentration oil will generate. Next, the emulsion which water is made to distribute this translucent concentration oil quickly, or becomes, and has a small grain size generates. GI (Gee) I hear that the fault of instruction technology must dilute a concentration oil with very a lot of water, and there is so that the last emulsion may hardly contain the silicone solid content which is many from about 5 % of the weight. GI (Gee) The average grain size of the prepared emulsion is usually less than 0.14 microns.

[0005] Although the micro emulsion of volatile silicone is taught in the industry (for example, U.S. Pat. No. 4,782,095 and 4,801,447), a lot of surfactants are required of these micro emulsions. A lot of surfactants needed with this conventional technology are detrimental for many uses. A clover check (Chrobaczek) and CHIDA (Tschida) At U.S. Pat. No. 5,057,572, they are a silicone fluid and GI (Gee). Manufacture of the polysiloxane replaced by the amino alkyl of heating a water-soluble emulsifier, water, and an acid at 50 degrees C in all symmetrically is taught. Needs,

such as the special sequence of a process process, for example, the turn of addition etc., are not taught to a clover check (Chrobaczek). Although [ according to instruction of a clover check (Chrobaczek) / this procedure ] an amino content can apply to the silicone fluid of 0.1meq (milliequivalent)/g, a micro emulsion is obtained in fact only in the amino content more than the threshold value of about 0.12 to 0.14 meq/g. The emulsion blooms cloudy, therefore the grain size of a low and an emulsion turns into grain size which is no longer a real micro emulsion from this threshold value. The true micro emulsion has somewhat the optical transparency expressed by less than about 150 ASTM \*\*\*\*\*.

[0006] In brain trust men's (Breneman) U.S. Pat. No. 5,234,495, preparing a micro emulsion by the method of blending the ORGANO denaturation polysiloxane, for example, an amino functionality polysiloxane, the ORGANO denaturation polysiloxane emulsifier, water, and an alkali-metal salt is taught. If high shearing mixing of the mixture is carried out at the same time it heats such a blend beyond the cloud point of mixture, when it cools, the liquid phase which can form a micro emulsion will arise.

[0007] A useful result will be obtained if the micro emulsion of amino functionality silicone especially a hyperviscous amino functionality silicone fluid, or gum is used for personal care product combination. Furthermore, MQ resin gives a desirable property to a personal care product. Moreover, a method of preparing the micro emulsion of the method of blending MQ resin with a personal care product and MQ resin is desired. To offer the substituting method or the improving method for preparing the micro emulsion of a small average grain size is desired as usual.

[0008]

[Summary of the Invention] One mode of this invention is a transparent oil-water-type micro emulsion containing the amino silicone fluid or gum which can be emulsified (a) detailed, (b) MQ (or siloxy silicate) resin or its mixture, a surfactant with high (c) phase inversion temperature, and (d) water.

[0009] another whole surface of this invention — average grain size — about 0.001– about 0.050 microns — desirable — about 0.010– about 0.030 microns — most — desirable — about 0.010– it is about 0.025 microns and the manufacture method of the transparent polyorganosiloxane micro emulsion containing the hyperviscous amino silicone fluid or gum which can be emulsified detailed, and at least one sort of surfactants with high phase inversion temperature is offered

[0010] Other aspects of affairs of this invention are the personal care products containing the micro emulsion of poly dimethylsiloxane-MQ resin mixture, and the micro emulsion of this invention.

[0011]

[Detailed description of invention] I hear that the organic-functions-ized silicone, for example, an amino functionality silicone fluid, and gum being able to form a micro emulsion, and the discovery used as the foundation of this invention being blended with a surfactant with high phase inversion temperature and this blend can be processed so that mixture may form a micro emulsion, and there are. When blended with other resins, especially MQ resin, it was discovered that the detailed emulsification of the amino functionality silicone-MQ resin blend can be carried out. Such a micro emulsion is usually transparent or translucent. "Transparency" means that there is not muddiness or cloudiness on these specifications, cloudiness is defined here by the ASTM examining method, especially the ASTM examining method D871 which uses a muddiness suspension standard, and cloudiness or muddiness makes about 150 an upper limit in this invention. If it blooms cloudy and \*\* exceeds about 50, the micro emulsion of this invention will begin to change to transparent shell order to translucence. The micro emulsion of this invention blooms cloudy, they are the ranges from 0 to about 150, and from 0 to about 50 is [ as for \*\*, from about 0 to about 80 is desirable, and ] the most desirable. The muddiness suspension standard used by this ASTM examining method D871 is U.S. New York State Garden City (Garden City). It is available from HERIGE (Hellige Incorporated). The cloudy scale of pure distilled water is 0.

[0012] They is about 0.005 – 0.050 microns of abbreviation, about 0.010 – 0.030 microns of abbreviation of the average grain size of the polyorganosiloxane micro emulsion prepared by the

method of this invention are desirable, and about 0.010 – its 0.025 microns of abbreviation are the most desirable. Generally, although cloudiness and average grain size are related mutually, all are influenced with the relative amount of three components with a main emulsion, i.e., silicon oil, the mixture of those, an emulsifier, and water. therefore, when the ratio of an oil and water is fixed, cloudiness and average grain size are related — although I will come out, unless other requirements are specified — as the index of the average grain size in a micro emulsion — blooming cloudy — the very thing — the need — enough — there are not conditions and a bird clapper

[0013] the term in this detailed in the letter one "detailed emulsification is possible" — the average grain size of an emulsion — 0.0001 — it means that a micro emulsion which is the range which is about 0.050 microns can be formed "The silicone which can be emulsified detailed" means the mixture of the silicone which can form the micro emulsion of the aforementioned definition, or silicone.

[0014] It is temperature equal [ to phase inversion temperature / to the lipophilic phase and hydrophilic phase in which a given surfactant exists simultaneously ] and meltable. or [ thinking generally ] — or the hydrophilic phase to be used is water In phase inversion temperature, a surfactant, a hydrophilic phase, and a lipophilic phase are in the minimum free-energy state thermodynamically. It is the feature that the grain size of the emulsion formed when this thermodynamics state emulsifies mixture is the minimum. That is, phase inversion temperature has a specific inclination to given component composition. Although phase inversion temperature changes with composition, when one of the two liquid phase, for example, water, is kept constant, the phase inversion temperature of a series of mixture using a given surfactant, water, and the various lipophilic phases of this water and immiscibility has the inclination to change by the much narrow temperature requirement.

[0015] It is performed as follows for preparing oil-resin-surfactant mixture in one mode of this invention.

(1) It is attached to the 100 sections of the last micro emulsion constituent, and blend the polyorganosiloxane of the amount of the range of the ten to 30 section most preferably the five to 40 section the one to 40 section. This polyorganosiloxane A (1) can be emulsified detailed, and depending on the case, amino contents are about 0.10 – about 3.0 milliequivalents /g, and it contains one or more sorts of silicone of the following formula.

[0016]  $M(R_a Q_b SiO(4-a-b)/2)_x y M(R_c SiO(4-c)/2)$  — here — the inside of a formula, and R — 1 — it is the hydrocarbon or hydrocarbon group which has about six carbon atoms, and Q is a polar group which has the following general formula

— R1 HZ, however R1 It is the bivalent connection machine combined with hydrogen and Basis Z, and it consists of a carbon atom and a hydrogen atom, consists of a carbon atom, a hydrogen atom, and an oxygen atom, or consists of a carbon atom, a hydrogen atom, and a nitrogen atom, and Z is the basis of the organic amino functionality containing at least one amino functional group. Moreover, for "a" in the above-mentioned formula, it is the ranges from about 0 to about 2, and "b" is the ranges from about 1 to about 3, however "a" + "b" is three or less, "c" is the number of the ranges from about 1 to about 3, and x is 1 to about 2, and 0. up to 00 — desirable — from about 3 up to about 50 — most — desirable — the number of the ranges from about 3 to about 25 — it is — y — from about 20 up to about 10,000 — desirable — from about 125 up to about 10,000 — most — desirable — the number of the ranges from about 150 to about 1,000 — it is — M — arbitration well-known in the industry — a suitable silicone end halt machine — it is trimethylsiloxy preferably As a non-limiting example of a basis expressed by R, they are alkyl groups (for example, a methyl, ethyl, a propyl, an isopropyl, butyl, an isobutyl, an amyl, an isoamyl, a hexyl, an iso hexyl, etc.). An alkenyl machine (for example, a vinyl, a halo vinyl, an alkyl vinyl, an allyl compound, a halo allyl compound, alkyl aryl), a cycloalkyl machine, phenyl groups (for example, cyclo butyl, cyclohexyl, etc.), a benzyl, halo hydrocarbon groups (for example, 3-chloropropyl, 4-BUROMO butyl, 3 and 3, 3-truffe RUORO propyl, chloro cyclohexyl, a BUROMO phenyl, chlorophenyl, etc.), and sulfur content machine s (for example, mercapto thyl, a mercapto propyl, a mercapto hexyl Desirable R is an alkyl group containing the carbon atom of 1 – six abbreviation, and most desirable R is a methyl. R1 As an example. \*\*, a methylene,

ethylene, a propyl ne, a hexamethylene, a deca methylene,  $-\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2-$ , Phenylene, Naphthylene,  $-\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2-$ ,  $-\text{CH}_2\text{CH}_2\text{OCH}_2-$ ,  $-\text{OCH}_2\text{CH}_2-$ ,  $-\text{OCH}_2\text{CH}_2\text{CH}_2-$ , and  $-\text{CH}_2\text{CH}(\text{CH}_3)\text{C}(\text{O})\text{OCH}_2-$ ,  $-(\text{CH}_2)\text{Ther}$  are 3  $\text{CC}(\text{O})\text{OCH}_2\text{CH}_2-$ ,  $-\text{C}_6\text{H}_4\text{C}_6\text{H}_4-$ ,  $-\text{C}_6\text{H}_4\text{CH}_2\text{C}_6\text{H}_4-$ , and  $-(\text{CH}_2)_3\text{C}(\text{O})\text{SCH}_2\text{CH}_2-$ .

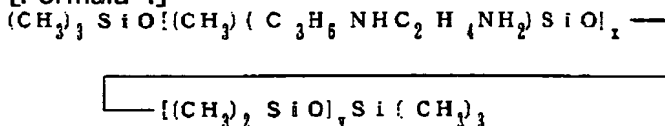
[0017] Z is the basis of the organic amino functionality containing at least one amino functional group. One formula possible as Z is  $-\text{NH}(\text{CH}_2)_z\text{NH}_2$ . However, z is one or more. Another formula possible as Z is  $-\text{N}(\text{CH}_2)_z\text{zzNH}(\text{CH}_2)_z$ . z and zz are one or more independently [ each ] here. This structure includes a diamino ring structure like piperazinyl one. Most desirable Z is  $-\text{NHCH}_2\text{CH}_2\text{NH}_2$ . It is a machine. Another formula possible as Z is  $-\text{N}(\text{CH}_2)_z\text{zzNX}(\text{CH}_2)_2$  or  $\text{NX}_2$ . It is  $\text{X}_2$  in a formula here. X is chosen from the groups to which each changes independently from hydrogen and the alkyl group of 1-12 carbon atoms, and zz is 0.

[0018] Q is formula  $-\text{CH}_2\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2$ . The polar group of the amine functionality which it has is the most desirable. "b" is the value of the ranges from about 1 to about 3, "a" is the value of the ranges from about 0 to about 2 by the aforementioned formula, and "c" is [ "a" + "b" is three or less and ] the number of the ranges from about 1 to about 3.  $\text{Ra Qb SiO} (4-a-b)/2$  A unit, and  $\text{Rc SiO} (4-c)/2$  the mole ratio of a unit — the ranges from about 1:2 to about 1:65 — it is — from about 1:5 up to about 1:65 — desirable — about — about [ 1:15 to ] — even 1:20 is the most desirable When using one or more sorts of silicone of the above-mentioned formula, the various substituents in the above-mentioned formula may differ among various silicone components containing silicone mixture.

[0019] As for the amino functionality silicone gum used by this invention, or fluid A (1), what has the following formula is desirable.

[0020]

[Formula 4]

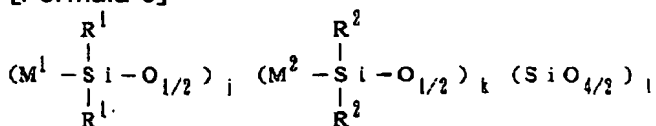


[0021] Here, x is the number of the ranges from about 3 to about 25 most preferably [ it is desirable from about 1 to about 2,000, and ] from about 3 to about 50, and y is the number of the ranges from about 150 to about 1,000 most preferably [ it is desirable from about 20 to about 10,000, and ] from about 125 to about 10,000. It is a range to 100 to 10,000,000 centistokes at 25 degrees C, the viscosity of this amino functionality silicone fluid or gum has the desirable viscosity of the range to 200 to 20,000 centistokes, and its viscosity of the range to 500 to 5,000 centistokes is the most desirable.

[0022] (2) It is attached to the 100 sections of the further last micro emulsion constituent, and MQ resin [ of the amount of the range of the 10 - 30 section ] A (2) is blended. This MQ resin has the following general formula (1).

[0023]

[Formula 5]



[0024] Here, it is  $\text{R}^1$ .  $\text{R}^2$  The alkyl group which all has 1-12 carbon atoms is sufficient, and it is  $\text{M}^1$ .  $\text{M}^2$  Each becomes independent, it is good in the arbitrary combination of the alkyl of a phenyl, a phenethyl, a polyether, hydrogen, or 1-23 carbon atoms, however Subscripts j, k, and l fill the next relation.

$$0.5 \leq (j+k) / l \leq 4.0 \quad (2)$$

This MQ resin is polymer which the illustrated compound kind which has the molecular weight of a certain range is distributed, and is constituted in itself. Setting the value of l as 1 can define MQ resin as a specific case. That is, at the time of  $l=1$ , the ranges of j are 0-4, and the ranges of k are 0-4. It is  $j+k=4$  at the time of  $l=1$  because of a stoichiometry-limit. Moreover, it is

desirable that the ratio of  $(j+k)/l$  is also equal to about 2. The relation of a formula (2) receives further limit that  $j+k$  must be at least 1. A desirable resin is  $R1 = CH3$ . It solves and the next value of Subscripts  $j$ ,  $k$ , and  $l$ ,  $j=2$  [ i.e., ],  $k=0$ , and  $l=1$  are satisfied.

[0025] M1 And/or, M2 When it is a polyether, the polyether has the following general formula.  
 $H2 C=C-CR3-(O-CHR4-CH2) u-(OCH2 CH2) v-OR5$  — here  $R3$  It is  $-(CH2) n-$  ( $n$  is the ranges from 1 to about 20),  $R4$  is the alkyl group of 1–20 carbon, and it is  $R5$ .  $H$  and  $-CH3$  And  $-C(O) CH3$  It is chosen from the groups which change, and  $v$  is the integer of the ranges from  $u$  and 0 to 20, however is  $u+v \geq 1$ . Notice the viscosity of MQ resin used for the cosmetics compound using the process of the micro emulsion constituent of this invention, and the micro emulsion of this invention, and the micro emulsion of this invention at 25 degrees C about it being a range to 100 to 2,000 centistokes most preferably [ it is desirable up to 50 to 5,000 centistokes, and ] up to 50 to 3,000 centistokes.

[0026] (3) It is attached to the last micro emulsion constituent 100 section, and add at least one sort of surfactant [ of the about one to 30 section ] A (4) to a process (1) and the silicone blend obtained by (2). Here, at least one sort of a surfactant has high phase inversion temperature, and, generally the phase inversion temperature is the range of about 45 – 95 degrees C of abbreviation.

(4) Heat to the temperature of the range of about 45 – 95 degrees C of abbreviation, agitating the blend of silicone and a surfactant. This temperature is made lower than the phase inversion temperature of a surfactant.

[0027] (5) Apply slowly only a weight equal to the weight of the silicone which used water (it considers as Water I) by Part I.

(6) Add the acid of the amount which sets the last pH of a micro emulsion to about 4–7. As for a process (5) and (6), it is desirable by adding water and a suitable acid separately or adding the solution of a suitable acid to carry out simultaneously. Although a desirable acid is an acetic acid, other acids like HCl, hypophosphorous acid, a lactic acid, a propionic acid, a glycolic acid, a formic acid, and a nitric acid can be used.

[0028] (7) Only the amount of the range of 40 – the about 90 sections adds water (it considers as Water II). From the temperature of the emulsion to which the temperature of this water II acidified, it is 0 – about 95-degree-C low range, and addition of this cold water will quench the temperature of the aforementioned micro emulsion. Furthermore, Water II may contain the polymer of the amounts of macromolecules, such as polyvinyl alcohol and a hydroxymethyl cellulose, in order to improve the stability of the last emulsion.

[0029] A (4) contains at least one sort of surfactants. Phase inversion temperature is the range which are 50 – 95 degrees C of abbreviation, and at least one sort of this surfactant calls this surfactant a primary surfactant henceforth. Other arbitrary surfactants are called secondary surfactant. Hydrophilic–lipophilic property balance values are about 10 – abbreviation 16, the blend of a surfactant or a surfactant has about 11 – a desirable abbreviation 16, and about 12 – its abbreviation 13 are the most desirable. When desirable hydrophilic – lipophilic property balance value increases the concentration of the volatile silicone in the silicone which can be emulsified detailed, it may change.

[0030] A primary surfactant is good by cation nature, anionic, Nonion nature, or amphoterism. The example of such a surfactant is GI (Gee). It is indicated by U.S. Pat. No. 4,620,878 (contained in this specification by quotation). For generally using it by this invention, the surfactant of Nonion nature is desirable. As a surfactant useful as a primary surfactant, it is at this invention. C10 which has ethyleneoxide to 95% – C22 fatty acid, or the polyethylene condensate of a fatty alcohol is in the fat amino \*\*\*\*\* amide betaine which has the polyoxyethylene sorbitan ester of C10 which has ethyleneoxide to 95% – C22 fatty acid, the polyoxyethylene sorbitol ester of C10 – C22 fatty acid, 6–20 carbon atoms, and the polyoxyethylene derivative of the fat phenol which has an ethyleneoxide to 95% and 10–22 carbon atoms, and a row.

[0031] As a primary surfactant desirable to operation of this invention This is a Nonion nature surfactant which has various quantity of an ethyleneoxide unit. alkyl phenoxy PORIETOKISHI ethanol [— Union Carbide (Union Carbide Corporation) By registered trademark called a shell

triton (TRITON), available ], It is polyethylene glycol-ether [Union Carbide () of the alcohol which is branching preferably and contains 11-15 carbon atoms although the trimethyl nonyl polyethylene-glycol ether and a line, or branching is sufficient. [ Union ] Carbide Corporation By registered trademark called shell Tergitol (TERGITOL), available], Although there is the ethoxylized tridecyl ether [available at a registered trademark called Emery Industries (Emery Industries) to a try call (TRYCOL)] of Nonion nature, it is not limited to these.

[0032] Although the trimethyl nonyl polyethylene-glycol ether and a line, or branching is sufficient as it, a surfactant desirable although it is used as a primary surfactant of this invention is branching preferably, and it is the polyethylene glycol ether of the alcohol containing 11-15 carbon atoms, and it is Union Carbide (Union Carbide Corporation). It is available at a registered trademark called shell Tergitol (TERGITOL). A surfactant desirable as a primary surfactant of this invention is the trimethyl nonyl polyethylene-glycol ether. The most desirable primary surfactant is a 2, 6, and 8-TORIMECHIRU-4-nonyloxy polyethylene oxide [Tergitol (TERGITOL) (registered trademark) TMN-6].

[0033] Arbitrary secondary surfactants may be good by anionic, cation nature, Nonion nature, or amphoterism, and fusibility or insolubility is sufficient as them at the desirable amino functionality silicone of A (1). The surfactant of Nonion nature is desirable. As a non-limiting example of a meltable surfactant, an alkylphenol ethoxy rate is in amino functionality silicone. Especially desirable arbitrary secondary surfactants are tritons (TRITON) chi-405 (registered trademark).

[0034] Moreover, as for the secondary surfactant arbitrarily used by this invention, it is desirable that it is also insoluble to the silicone of A (1). A surfactant desirable although it is used as a secondary surfactant by this invention is alkyl phenoxy PORIETOKISHI ethanol. per [ 1 / about ] 100 weight sections of the micro emulsion constituent of last / amount / of A (4) ] - the about 30 weight section — desirable — about 1 — the about 20 weight section — it is the range of about 5 — the about 15 weight section most preferably

[0035] The blend of silicone, a surfactant, and water is homogenized with the suitable mixed equipment of a homogenizer and others. Depending on a mixed device parameter, this contractor can decide on time required at this process to form homogeneous mixture or a homogeneous emulsion, without conducting too much experiment. In order to form the micro emulsion of this invention, high shearing mixing under the conditions by which an ordinary pressure or a reaction medium is pressurized is not usually required. Since the blend contains the surfactant with high phase inversion temperature, you have to adjust carefully the temperature in which a micro emulsion is formed. therefore, the process which adds Water I — a 45-95-degree C temperature requirement — desirable — a 55-90-degree C temperature requirement — it carries out by th 65-85-degree C temperature requirement most preferably

[0036] a process (6) — a micro emulsion — acidifying — pH of an emulsion — 4-7 — desirable — 5-6.5 — it carries out within the limits of 5.5-6.5 most preferably Especially if this process is combined with a process (5), it is effective. In order to change pH of a reaction medium, it is necessary to take into consideration the amount of the amino functionality silicone which exists in a reaction medium, or silicone. It depends on the amount of amino functionality silicone or silicone fluid A (1), and the amino content of an amino functionality silicone fluid for the amount of the acid needed for obtaining such pH value. For example, when the amino contents of an amino functionality silicone fluid are 0.6 milliequivalents /g, the amount of enough acids to make pH into request within the limits is attached to the 100 weight sections of an amino functionality silicone fluid, and is the about 2.5 weight section. When the amino functionality silicone fluid has the amino content of 3.0 milliequivalents / g, the weight of an acid is attached to the fluid 100 weight section, and is the about 1.25 weight section. Although it may change depending on the molecular weight or the equivalent of an acid chosen as the weight of an acid required to attain given pH controlling pH, it is the main purpose of acid addition to adjust pH to a desired value. Furthermore, that it must be simultaneous with addition of Water I has made addition of an acid clear.

[0037] The amino functionality silicone micro emulsion of this invention is useful for various personal care product uses like compounds for hair fixation, such as a hair conditioner, the so-called Tween-one (2-in-1) shampoo, and a style gel mousse. the conditioner compound of the



purpose of a personal care use — usually — about 1– about 20 % of the weight — desirable — about 5– about 10 % of the weight — most — desirable — about 6– it has the amino functionality silicone micro emulsion content of about 7% of the weight of the range The amino functionality silicone micro emulsion content of the Tween-one shampoo compound of the purpose of a personal care use is [ about 0.5 – 10 % of the weight of abbreviation ] usually the range of about 3 – 4 % of the weight of abbreviation most preferably about 1 – 5 % of the weight of abbreviation. The amino functionality silicone micro emulsion content of the compound for fixation of the purpose of a personal care use is [ about 0.5 – 10 % of the weight of abbreviation ] usually the range of about 3 – 5 % of the weight of abbreviation most preferably about 2 – 6 % of the weight of abbreviation. The personal care product using the micro emulsion prepared by the method of this invention usually blooms cloudy, and \*\* is about 100 low. The range of weight % used on these specifications above is a weight % range over the last micro emulsion as one component of a personal care product. That is, the silicone content of the micro emulsion prepared by the method of this invention changes at about 0.5 – 25 % of the weight of abbreviation, and this changes as % of the last composition of a personal care product at about 0.1 – 7 % of the weight of abbreviation, when this micro emulsion is blended into a personal care product. Furthermore, the micro emulsion of this invention can be blended with a textile processing product or a skin care compound, for example, coloring cosmetics.

[0038] All U.S. patents quoted here shall be included in this specification by having quoted.

[0039]

[Example]

Fruit The constituent of the following non-limiting examples 1–24, i.e., the examples which illustrate the micro emulsion of this invention, was prepared using the procedure which indicated the outline in the column of detailed explanation of \*\*\*\*\*. The example which uses these micro emulsions for a personal care product is also shown. Examples 39–43 are instantiation of a personal care compound.

[0040] an example — 170 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — — (CH<sub>2</sub>) — three — — — NH-CH — two — — — CH — two — — — NH — two — viscosity — 150 — — — 400 — centistokes — an amine — a content — 0.12 — meq/g —] — 16 — the section — Tergitol (TERGITOL) TMN-6 (eight sections) — having mixed — Next, Water I (16 sections) was dropped and added at 70 degrees C. It is 1 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened and became translucent. Next, it added quickly, often stirring Water II (58 sections). When it cooled, the micro emulsion of about 40 was obtained for ASTM \*\*\*\*\*.

[0041] The constituent of the example of one example of comparison was prepared like the example 6 of U.S. Pat. No. 5,057,572. 70 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — — (CH<sub>2</sub>) — three — — — NH-CH — two — — — CH — two — — — NH — two — viscosity — 150 — — — 400 — centistokes — an amine — a content — 0.12 — meq/g —] — 16 — the section — Tergitol (TERGITOL) TMN-6 (eight sections) and water (74 sections) — Next, the lactic acid (one section) was added at 70 degrees C. The after [ cooling ] milk-like emulsion was obtained. This manufacture object bloomed cloudy and \*\* was larger than 200.

[0042] Example 2 this example shows the effect when adding Water II slowly. 70 — degree C — slight tepidness — \*\*\*\*\* — amino ones — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — — (CH<sub>2</sub>) — three — — — NH-CH — two — — — CH — two — — — NH — two — — viscosity — 150 — 400 — centistokes — an amine — a content — 0.12 — meq/g —] — 20 — — the section — Tergitol (TERGITOL) TMN-6 (12.5 sections) — having mixed . Next, Water I was dropped and added at 70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened and became translucent. Next, it dropped and added, often stirring Water II (53 sections). It thickened slowly, water became near the addition end after that, and mixture became thin. When it cooled, it bloomed cloudy and the micro emulsion of about 200 was obtained for \*\*.

[0043] Three to example 7 this example shows the effect by the difference in the amount of

Water I (the displayed amount is a gram).

Example No 3 4 5 6 7 \*\* Part Amino fluid A1 65 65 65 65 65 Tergitol TMN-6 40 40 40 40 40  
Water I 40 65 90 120 220 Acetic acid 1 1 1 1 1 Water II 180 155 1130 100 0 It blooms cloudy.  
150 30 90 200+ 200+pH 5.5 5.5 5.5 5.5 Effect according to the difference in the amount of a  
surfactant at eight to nm example 12 this example. It is shown (the displayed amount is a gram).  
[0044]

Example No 8 9 10 11 12 \*\* Part Amino fluid A1 65 65 65 65 65 Tergitol TMN-6 65 40 20 45  
27.5 Water I 65 65 65 65 65 Acetic acid 1 1 1 1 1 Water II 130 155 175 150 167.5 It blooms  
cloudy. 100 40 200 80 100 pH 5.55.5 5.5 5.5 Effect by the difference which is the effect and  
amino fluid by difference of temperature of Water II in 13 to 5.5 example 17 this example. It is  
shown (the displayed amount is a gram).  
[0045]

Example No 13 14 15 16 17 \*\* Part Amino fluid A1 65 65 65 0 0 Amino fluid A2 0 0 0 65 65  
Tergitol TMN-6 40 40 40 40 40 Water I 65 65 65 65 65 Acetic acid 1 1 1 1 1 Water II 155 155115  
155 0 Water II temperature \*\* 25 075 25 It blooms cloudy 25. 4050 50 15 30 pH 5.5 5.5 5.55.5  
Amine concentration which is different in 18 to nm example 24 this example with a low amino  
content. The effect when using the fluid which it has is shown (the displayed amount is a gram).  
[0046]

Example No 18 19 20 21 22. 23 24 \*\* Part The amino fluid A1 0 50 0 0 0 0 0 The amino fluid A3  
65 15 25 15 0 0 0 The amino fluid A4 0 0 40 50 65 40 65 Amino fluid A5 0 0 0 0 0 25 0 Tergitol  
TMN-6 40 40 40 40 40 40 40 Water I 65 65 6565 65 65220 Acetic acid 1 11 11 11 Water II 155  
155155 155 155155 0 Cloudiness 200+ 100 80 50 40 60 60 pH 5.5 5.5 5.5 5.5 5.5 5.5 A3 =  
[ Line, ] They are an end halt,  $-(CH_2)_3-NH-CH_2-CH_2-NH_2$ , viscosity =400 centistokes, and  
amine content 0.07 meq/g at trimethylsilyl.

A4 = they are an end halt,  $-(CH_2)_3-NH-CH_2-CH_2-NH_2$ , 4500 to viscosity =5000 centistokes,  
and amine content 0.12 meq/g at a line and trimethylsilyl.

A5 = they are an end halt,  $-(CH_2)_3-NH-CH_2-CH_2-NH_2$ , 4500 to viscosity =5000 centistokes,  
and amine content 0.07 meq/g at a line and trimethylsilyl.

[0047] an example — 2570 — degree C — slight tepidness — \*\*\*\*\* — amino one —  
functionality — silicone — [— a line — trimethylsilyl — an end — a halt —  $(CH_2)$  — three —  
NH-CH — two — CH — two — NH — two — a pendant — viscosity — 270,000 — centistokes —  
— an amine — a content — 0.6 — meq/g —] — 20 — the section — Tergitol (TERGITOL)  
TMN-6 (12.5 sections) — having mixed . Next, Water I (20 sections) was dropped and added at  
70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of  
water. Mixture was thickened quickly and became translucent. Next, it added quickly, often  
stirring Water II (47 sections). The micro emulsion of about 40 was obtained for ASTM \*\*\*\*\*.  
When heating aging was carried out for two weeks at 50 degrees C, it bloomed cloudy, and \*\*  
increased to 50.

[0048] Example 26 this example shows the effect when adding the buffer solution to an emulsion  
compound. 70 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality —  
silicone — [— a line — trimethylsilyl — an end — a halt —  $(CH_2)$  — three — NH-CH — two —  
CH — two — NH — two — a pendant — viscosity — 270,000 — centistokes — an amine — a  
content — 0.6 — meq/g —] — 20 — the section — Tergitol (TERGITOL) TMN-6 (12.5  
sections) — having mixed . Next, Water I (20 sections) was dropped and added at 70 degrees C.  
The solution of the after [ the completion of addition ] water 2 section of water, the acetic-acid  
0.5 section, and the sodium acetate 0.25 section was added. Mixture was thickened quickly and  
became translucent. Next, it added quickly, often stirring Water II (44.75 sections). The micro  
emulsion of about 10 was obtained for ASTM \*\*\*\*\*. When heating aging was carried out for two  
weeks at 50 degrees C, it bloomed cloudy, and \*\* increased to 40.

[0049] Example 27 this example shows the effect when changing the amount of Water I. 70 —  
degree C — slight tepidness — \*\*\*\*\* — amino ones — functionality — silicone — [— a lin —  
— trimethylsilyl — an end — a halt —  $(CH_2)$  — three — NH-CH — two — CH — two — NH — two  
— viscosity — 270,000 — centistokes — an amine — a content — 0.6 — meq/g —] — 20 —  
the section — Tergitol (TERGITOL) TMN-6 (12.5 sections) — having mixed . Next, Water I (24

sections) was dropped and added at 70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened quickly and became translucent. From the mixture obtained in the example 1, this mixture is not viscous and tended [ much ] to work. Next, it added quickly, often stirring Water II (47 sections). The micro emulsion with ASTM \*\*\*\*\* smaller than 10 was obtained. When heating aging was carried out for two weeks at 50 degrees C, it bloomed cloudy, and \*\* increased to 40.

[0050] Example 28 this example shows the effect when changing the amount of Water I. 70 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — (CH<sub>2</sub>) — three — NH-CH — two — CH — two — NH — two — a pendant — viscosity — 270,000 — centistokes — an amine — a content — 0.6 — meq/g — ] — 20 — the section — Tergitol (TERGITOL) TMN-6 (12.5 sections) — having mixed . Next, Water I (ten sections) was dropped and added at 70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened quickly and became translucent. This emulsion was not easily stirred with a viscous past \*\* overhead stirrer. Next, it added quickly, often stirring Water II (57 sections). The micro emulsion of about 20 was obtained for ASTM \*\*\*\*\* . The emulsion was divided into the bilayer when heating aging was carried out for two weeks at 50 degrees C.

[0051] Example 29 this example shows the effect when using the blend of a surfactant. 70 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — (CH<sub>2</sub>) — three — — — NH-CH — two — — — CH — two — — — NH — two — a pendant — viscosity — 270,000 — centistokes — an amine — a content — 0.6 — meq/g — ] — 20 — the section — Tergitol (TERGITOL) TMN-6 (nine sections) and a triton (TRITON) chi- Next, Water I (24 sections) was dropped and added at 70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened quickly and became translucent. Next, it added quickly, often stirring Water II (43.5 sections). The micro emulsion of about 50 was obtained for ASTM \*\*\*\*\* . When heating aging was carried out for two weeks at 50 degrees C, it bloomed cloudy, and \*\* increased to 100.

[0052] Example 30 this example shows the effect when using the blend of a surfactant. 70 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — (CH<sub>2</sub>) — three — — — NH-CH — two — — — CH — two — — — NH — two — a pendant — viscosity — 270,000 — centistokes — an amine — a content — 0.6 — meq/g — ] — 20 — the section — Tergitol (TERGITOL) TMN-6 (1.1 sections) and a triton (TRITON) chi- Next, Water I (24 sections) was dropped and added at 70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened quickly and became translucent. Next, it added quickly, often stirring Water II (43.5 sections). The micro emulsion with ASTM \*\*\*\*\* smaller than 10 was obtained. When heating aging was carried out for two weeks at 50 degrees C, it bloomed cloudy, and \*\* was conspicuous and did not increase.

[0053] Example 31 this example shows the effect when using the blend of a surfactant. 70 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — (CH<sub>2</sub>) — three — — — NH-CH — two — — — CH — two — — — NH — two — viscosity — 270,000 — centistokes — an amine — a content — 0.6 — meq/g — ] — 20 — the section — Tergitol (TERGITOL) TMN-6 (8.5 sections) and a triton (TRITON) chi-405 (1.5 Next, Water I (24 sections) was dropped and added at 70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened quickly and became translucent. Next, it added quickly, often stirring Water II (43.5 sections). The emulsion with larger ASTM \*\*\*\*\* than 100 was obtained. When this emulsion carried out heating aging at 50 degrees C, it was separated.

[0054] Example 32 this example shows the effect when changing amino functionality silicone. 70 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — (CH<sub>2</sub>) — three — NH-CH — two — CH — two — NH — two — viscosity — 140,000 — centistokes — an amine — a content — 0.3 — meq/g — ] — 20 — the section — Tergitol (TERGITOL) TMN-6 (12.5 sections) — having mixed . Next, Water I

(20 sections) was dropped and added at 70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened quickly and became translucent. Next, it added quickly, often stirring Water II (47 sections). The micro emulsion of about 10 was obtained for ASTM \*\*\*\*\*. When heating aging of this emulsion was carried out for two weeks at 50 degrees C, it separated into the bilayer.

[0055] Example 33 this example shows the effect when using a different fluid. 70 — degree C — slight tepidness — \*\*\*\*\* — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — — (CH<sub>2</sub>) — three — — — NH-CH — two — — — CH — two — — — NH — two — a pendant — viscosity — 140,000 — centistokes — an amine — a content — 0.3 — meq/g —] — 20 — the section — Tergitol (TERGITOL) TMN-6 (11 sections) and a triton (TRITON) chi- Next, Water I (24 sections) was dropped and added at 70 degrees C. They are 0.5 \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened quickly and became translucent. Next, it added quickly, often stirring Water II (43.5 sections). The micro emulsion with ASTM \*\*\*\*\* smaller than 10 was obtained. When heating aging was carried out for two weeks at 50 degrees C, it bloomed cloudy, and \*\* was conspicuous and did not increase.

[0056] 34 to example 38 examples 34-38 were prepared with the technology of a publication in the example 25. The result obtained about the manufacture object illustrated in the examples 34-38 is collectively shown in the next table.

Example No 34 35 36 37 38. Fluid A 20 20 0 0 0 Fluid B 0 0 20 20 0 Fluid C 0 0 0 0 20 Primary surfactant a 12.5 8.5 12.5 8.5 12.5 Secondary surfactant b 0 2.5 0 2.50 Water I 2525 2525 The 25 buffer solution 3.73.7 3.03.0 2.5 water II 38.8 40.3 39.541 It blooms cloudy in early stages of 40. 2020 3030 40 It blooms cloudy after aging. 40 20 50 40 >100 fluid A = they are an end halt, — (CH<sub>2</sub>)<sub>3</sub>-NH-CH<sub>2</sub>-CH<sub>2</sub>-NH<sub>2</sub>, viscosity 2,000,000 centistokes, and amine content 0.6 meq/g at a line and trimethylsilyl.

Fluid B = they are an end halt, —(CH<sub>2</sub>)<sub>3</sub>-NH-CH<sub>2</sub>-CH<sub>2</sub>-NH<sub>2</sub>, viscosity 1,500,000csk, and amine content 0.3 meq/g at a line and trimethylsilyl.

Fluid C = they are an end halt, —(CH<sub>2</sub>)<sub>3</sub>-NH-CH<sub>2</sub>-CH<sub>2</sub>-NH<sub>2</sub>, viscosity 800,000csk, and amine content 0.15 meq/g at a line and trimethylsilyl. Primary surfactant = Tergitol (TERGITOL) TMN-6 and Union Carbide (Union Carbide) Make.

Secondary surfactant = a triton (TRITON) chi-405 and Union Carbide (Union Carbide) Make.

A buffer solution is the water 3 section, the acetic-acid 0.5 section, and the sodium acetate 0.2 section. 50-degree C oven performed aging for three weeks among the bottom airtight container of nitrogen.

[0057] Examples 39-43: The personal care product illustrated in the personal care product combination examples 39-43 was prepared using the micro emulsion (20 % of the weight of silicone contents) prepared by the method of this invention by trimethylsilyl using the linear amino functionality silicone which the end stopped. In the examples 39 and 40, the viscosity of 25 degrees C used the micro emulsion in which an amino content contains the silicone fluid of 0.55 meq/g by 150 centistokes. In the examples 41-43, the viscosity of 25 degrees C used the micro emulsion in which an amino content contains the hyperviscous amino silicone fluid of 0.30 meq/g by 189,000 centistokes.

[0058] The transparent conditioner was prepared from the component of the 39th example.

\*\* Charge Amount (% of the weight) Deionized water . 88.55 Hydroxyethyl cellulose 1.0 Chlorination SETORIMONIUMU 3.5 Silicone micro emulsion 6.0 Gley DANTO plus (Glydant Plus) (registered trademark) 0.2 Perfume The manufacture procedure used 0.75 times is as follows.

[0059] It added to deionized water, often stirring a hydroxyethyl cellulose. It stirred until it added gley DANTO plus (Glydant Plus) (registered trademark) and mixture became transparent again, when distributing completely. This water mixture was heated at 60 degrees C. When mixture was transparent, the silicone micro emulsion prepared by the method of chlorination SETORIMONIUMU and this invention was added separately. It cooled radiationally stirring mixture. Perfume was added when temperature became 40 degrees C or less. Churning was continued for about 20 minutes after addition of perfume.

[0060] Generally, an arbitrary component can be changed, replaced or omitted according to

instruction of the industry. For example, a preservative can be added in order to suppress bacterial multiplication. Furthermore, perfume, a pH regulator, an antistatic agent or a softener, cation nature polymer, a thickener, Nonion nature polymer, for example, acrylic-acid polymer, a neutralizer, for example, a triethanolamine, sunscreen, an antioxidant, protein, a vitamin, a vegetable extract, etc. can be added.

[0061] Conditioning or the Tween-one shampoo was prepared from the component of the 40th example.

\*\* Charge Amount (% of the weight) Deionized water 33.89 Hydroxyethyl cellulose 2.0 Lauryl ammonium sulfate (as 26% solution) 15.38 Laureth (Laureth) Ammonium sulfate 21.43 (as 28% solution)

Cocamide propyl betaine (as 35% solution) 11.43 Dowicil 200 (registered trademark) (Dowicil) 0.2 Silicone micro emulsion 5.0 Citric acid It adjusts to desired pH. It carries out. Sufficient amount RAURIN amide DEA 3.5 Chlorination SETORIMONIUMU (Cetrimonium Chloride) 6.67 Perfume The manufacture procedure used 0.5 times is as follows.

[0062] The hydroxyethyl cellulose was added to water, and it stirred until the hydroxyethyl cellulose fully carried out the solvation. Dowicil (Dowicil) 200 (registered trademark) was added, water mixture was stirred, and it heated at 60 degrees C. It stirred until mixture came to have presented uniform appearance, after adding to the order which showed the surfactant above, respectively. RAURIN amide DEA was dissolved and it added to mixture. Next, it cooled stirring mixture and churning between cooling was continued. It stirred, after adding a silicone micro emulsion and perfume separately in the place where temperature became lower than 40 degrees C. After adding the last component, mixture was continued for about 20 minutes.

[0063] The transparent conditioning shampoo compound was prepared from the component of the 41st example.

\*\* Charge Weight % deionized water 31.32 Lauryl ammonium sulfate (as 26% solution) 24.00 Laureth (Laureth) Ammonium sulfate 14.30 (as 28% solution)

Cocamide propyl betaine (as 35% solution) 11.43 An amino silicone GAMUMIKURO emulsion 7.0 Cocamide MEA (Cocamide) 2.5 A polysorbate 80 (Polysorbate) 2.5 The RAURIN amide DEA 2.0 A glycerol 2.0 Dimethicone copolyol 1.0 (Dimethicone Copolyol) A guar hydroxypropyl trimonium chloride 0.75 Perfume 0.75 FD & C yellow #5 (1.0% solution) 0.25 Methyl paraben (Methyl Paraben) 0.15 Propylparaben 0.05 methyl paraben and the propylparaben were melted in water. It added slowly, often stirring a guar hydroxypropyl trimonium chloride (guar hydroxypropyltrimonium chloride) in this solution. The lauryl ammonium sulfate, the laureth ammonium sulfate, and the cocamide propyl betaine (cocamidopropyl betaine) were added to this order in the place as for which this guar compound fully carried out the solvation, respectively. The solution was heated at 65 degrees C. It added having dissolved the RAURIN amide DEA and Cocamide MEA together, and often stirring them in a hot solution. Heating was stopped. Desired perfume was mixed together in the polysorbate 80, the glycerol, the silicone fluid, and the coloring agent row, and after temperature cooled at 45 degrees C or less, it added to the solution. After continuing churning for further 10 – 15 minutes, the silicone gum micro emulsion was added. Churning was continued for 15 more minutes after addition of a silicone gum micro emulsion.

[0064] The hair conditioner compound was prepared from the component of the 42nd example.

\*\* Charge Weight % Deionized water 73.40 A silicone gum micro emulsion 10.00 Cyclomethicone (Cyclomethicone) 5.00 a BEHEN trimonium methosulfate — and 2.75 Cetearyl alcohol A glycerol 2.50 Dimethicone copolyol 1.75 (Dimethicone Copolyol) Stearoamide propyl dimethylamine 1.50 Cetyl alcohol 1.50 Pentaerythritol tetrapod stearate 1.30 methylparaben (Methyl Paraben) 0.20 Propylparaben 0.10 Perfume The amount methylparaben of requests, and propylparaben. It melted in water. The solution was heated at 65 degrees C. It added to the solution, having dissolved BEHEN trimonium methosulfate (behentrimonium methosulfate) cetearyl (reaching) alcohol (cetearyl alcohol), a stearoamide propyl dimethylamine, and pentaerythritol tetrapod stearate together, and often stirring them. The solution was cooled radiationally. A glycerol and dimethicone copolyol were mixed together with arbitrary perfume, and after temperature fell to 45 degrees C or less, it added to the solution. Churning was continued for 15 – 20 minutes. The

silicone gum micro emulsion was added and it stirred for further 10 – 15 minutes.

Cyclomethicone was added and it stirred for 10 more minutes.

[0065] The hair conditioning style mousse was prepared from the component of the 43rd example.

\*\* Charge Weight % Deionized water 79.45 Poly quarter NIUMU 11 (Polyquaternium) 5.80 Oleth – 20 (Oleth) 0.75 A silicone gum micro emulsion 1.50 Dimethicone copolyol 0.50 (Dimethicone Copolyol) Hydrocarbon system injection agent Until it heats 12.00 water at 45 degrees C, it adds poly quarter NIUMU 11 and it melts completely. The solution was stirred. Next, Oleth –20 was added, and it continued stirring until it dissolved completely. The solution was cooled at 45 degrees C or less after that, and a silicone gum micro emulsion and dimethicone copolyol were added. Churning was continued until the solution got cold. The pressurization container was filled up with this liquid mousse compound after that, and it was pressurized with light hydrocarbon mixture.

[0066] Generally the above example explained the manufacture and the use of a micro emulsion which were prepared from the single amino silicone fluid. The viscosity of an amino silicone fluid can be changed broadly. The following examples explain the use to the micro emulsion and its personal care constituent of the mixture of an amino functionality silicone fluid, combination of MQ resin and MQ resin obtained as a result, and amino functionality silicone.

[0067] Example 44: Blend emulsification of MQ amino functionality silicone resin blend, general procedure 1 amino functionality silicone A (1), and MQ resin A (2).

2) Apply preferably surfactant A (3) and the secondary–alcohol ethoxy rate branched preferably by the ratio of 1.0:0.625 (an oil / resin mixture : a surfactant, a weight ratio).

[0068] 3) Blend at 70–75 degrees C.

4) Add slowly, stirring the water I of an amount equal to the amount of an oil, with the temperature of 70 degree C maintained.

5) an acid — add an acetic acid preferably and adjust pH

6) Add Water II quickly. This water may contain the fusibility component of an addition like the additive of a biocide and others.

[0069] Example 45: The amino content carried out the detailed emulsification of the solution of 50% of the weight of a methyl MQ resin with the amino silicone fluid of 0.8 meq/g, and the trimethylsilyl amino dimethicone among the personal care product cyclomethicone (cyclomethicone) using MQ amino functionality silicone blend which carried out detailed emulsification. The amino silicone fluid was [ MQ solution in cyclomethicone of the weight ratio of the mixture of silicone ] 50% 50%. The conditioning KARURI fresher who has the following composition using this micro emulsion was prepared.

[0070]

\*\* Charge Weight % Deionized water 89.99 Poly quarter NIUMU 11 (Polyquaternium) 0.67 Aminomethyl propanol 0.03 A polysorbate –80 (Polysorbate) 0.60 Dimethicone copolyol 0.50 (Dimethicone Copolyol) Micro emulsion mixture 5.00 Glycerol 2.50 A citric acid 0.01 The methylparaben 0.15 (Methyl Paraben) A propylparaben 0.05 (Propyl Paraben) Perfume Among the personal care product cyclomethicone (cyclomethicone) using MQ amino functionality silicone blend which carried out 0.50 Example 46:detailed emulsification, the solution of 50% of the weight of the methyl MQ An amino content is the amino silicone fluid of 0.8 meq/g, and the trimethylsilyl amino dimethicone. Detailed emulsification was carried out. The amino silicone fluid was [ MQ solution in cyclomethicone of the weight ratio of the mixture of silicone ] 50% 50%. The skin care product which has the following composition using this micro emulsion was prepared.

[0071]

\*\* Charge Weight % . Deionized water 79.90 Cetyl alcohol 2.00 glyceryl stearate — and 2.50 PEG–100 stearate Acetylation lanolin alcohol 2.00 The dimethicone 3.00 Micro emulsion mixture 8.00 Magnesium aluminum silicate 1.50 Perfume 0.50 propylparabens (Propyl Paraben) 0.10 Butylparaben (Butyl Paraben) 0.15 Methylparaben (MethylParaben) 0.15 phenoxyethanol To general 0.20, the arbitrary component in a personal care product can be changed, replaced or omitted according to instruction of the industry. For example, a preservative can be added in order to suppress bacterial proliferation. Furthermore, perfume, a pH regulator, an antistatic

agent or a softener, cation nature polymer, a thickener, Nonion nature polymer, for example, acrylic-acid polymer, a neutralizer, for example, a triethanolamine, sunscreen, an antioxidant, protein, a vitamin, vegetable extractives, etc. can be added.

[0072] an example — 47 — : — MQ — micro — an emulsion — manufacture — amino one — functionality — silicone — [— a line — trimethylsilyl — an end — a halt — — (CH<sub>2</sub>) — three — — NH—CH — two — — NH — two — a pendant — viscosity — 1,500 — — 2,500 — csk — an amine — a content — 0.8 — meq/g —] — 15 — a weight — the section — the mixture 5 weight section of the 1:1-fold quantitative ratio of Mixture was heated at 65 degrees C. Next, it added slowly, often stirring heating the water 20 weight section at 70 degrees C. It is 0.5 weight \*\*\*\*\* about the acetic acid after the completion of addition of water. Mixture was thickened and became transparent substantially. It added quickly, often stirring Water II (47.5 weight sections), after stirring for 2 – 5 minutes at 70 degrees C. When it cooled, the micro emulsion of 20 was obtained for ASTM \*\*\*\*\*.

[0073] this example explains the type of the fluid and resin which form a micro emulsion. Experimental run number 48 49 50 51 52 53 54 55 56 Component (weight section per 100 weight sections)

Fluid A1 15 15. 15 10 10 0 0 0 0 Fluid A2 0 0 0 0 0 15 15 12.5 10 Resin R1 5 0 0 10 0 5 0 0 10 Resin R2 0 5 0 0 10 0 5 7.5 0 Resin R3 0 0 5 0 0 0 0 0 0 TMN-612 12 12 12 12 12 12 12 12 Water I 20 20 20 20 20 20 20 20 20 Acetic acid 0.5 0.5 0.5 0.5 0.5 0.5 0.5 0.5 0.5 Water II 47.5 47.5 47.5 47.5 47.5 47.5 47.5 47.5 47.5 ASTM cloudiness 10 10 10 20 20 10 20 20 200+ Experimental run number 57 58 59 60 61 62 63 64 65 Component (weight section per 100 weight sections) Fluid A2 0 0. 0 0 0 0 15 15 15 Fluid A3 15 15 0 0 0 0 0 0 0 Fluid A4 0 0 17.5 17.5 0 0 0 0 0 Fluid A5 0 0 0 0 15 17.5 0 0 0 Resin R1 50 2.5 50 2.5 00 0 Resin R2 0 5 0 0 5 0 5 50 Resin R3 00 00 00 00 5 TMN-6 1212 12 12 12 12 00 12 15-S-9 0 00 0 00 12 00 (registered trademark) MINFOMU 0 0 0 0 0 0 0 12 0 (Minfoam) -1chi (registered trademark) Water I 20 20. 20 20 20 20 20 20 20 Acetic Acid 0.5 0.5 0.5 0.5 0.5 0.5 0.5 0.5 0.5 Water II 47.5 47.5 47.5 47.5 47.5 47.5 47.5 47.5 47.5 ASTM cloudiness 20 20 60 100+ 200+ 200+ 20200+ 20 Experimental run number 66 67 68 69 70 71 72 73 74 Component (weight section per 100 weight sections)

Fluid A1 17.5 0. 15 15 17.5 17.5 0 0 17.5 Fluid A2 0 17.5 0 0 0 0 17.5 17.5 0 Resin R4 2.5 2.5 0 0 2.5 0 0 0 0 Resin R5 0 0 5 0 0 0 0 0 0 Resin R6 0 0 0 5 0 0 0 0 0 Resin R7 0 0 0 0 0 2. 5 2.5 00 Resin R8 0 0 0 0 0 0 0 2.5 2.5 TMN-6 12 12 12 12 12 12 12 12 12 water I 20 20 20 20 20 20 20 20 20 Acetic acid 0.5 0.5 0.5 0.5 0.5 0.5 0.5 0.5 0.5 Water II 47.5 47.5 47.5 47.5 47.5 47.5 47.5 47.5 47.5 ASTM cloudiness 200+200+ 200+ 200+ 200+ 200+200+ 200+ 200+ fluids A1 Line, They are an end halt and —(CH<sub>2</sub>)<sub>3</sub>—NH—(CH<sub>2</sub>)<sub>2</sub>—NH<sub>2</sub> at trimethylsilyl. It is the silicone of a pendant, viscosity 1,500 – 2,500csk, and amine content 0.8 meq/g.

[0074] A fluid A2 is an end halt and —(CH<sub>2</sub>)<sub>3</sub>—NH—(CH<sub>2</sub>)<sub>2</sub>—NH<sub>2</sub> at branching and trimethylsilyl. It is the silicone of a pendant, viscosity 150 – 300csk, and amine content 0.53 meq/g. A fluid A3 is an end halt and —(CH<sub>2</sub>)<sub>3</sub>—NH—(CH<sub>2</sub>)<sub>2</sub>—NH<sub>2</sub> at a line and trimethylsilyl. It is the silicone of a pendant, viscosity 60,000csk, and amine content 0.3 meq/g.

[0075] A fluid A4 is an end halt and —(CH<sub>2</sub>)<sub>3</sub>—NH—(CH<sub>2</sub>)<sub>2</sub>—NH<sub>2</sub> at branching and trimethylsilyl. It is the silicone of a pendant, viscosity 150 – 300csk, and amine content 0.15 meq/g. Fluid A5 is an end halt and —(CH<sub>2</sub>)<sub>3</sub>—NH—(CH<sub>2</sub>)<sub>2</sub>—NH<sub>2</sub> at branching and trimethoxy one. It is the silicone of a pendant, viscosity 10 – 40csk, and amine content 0.45 meq/g.

[0076] a resin R1 — 1:1 (weight) mixture of volatile silicone (D5) and MQ methyl resin — it is — a M/Q ratio — it is =0.76 a resin R2 — 1:1 (weight) mixture of poly dimethylsiloxane and MQ methyl resin — it is — a M/Q ratio — it is =0.76 A resin R3 is the dryness article of a resin R1, and a silicone solid content is 100%.

[0077] The ratio of a phenyl pair methyl is TD resin of about 1:1, and a resin R4 is about 85% solid content in naphthalene. The ratio of a phenyl pair methyl is TD resin of about 1:1, and a resin R5 is about 50% solid content in toluene. The ratio of a phenyl pair methyl is TD resin of 2:3, and a resin R6 is about 80% solid content in solvent mixture of toluene and isopropyl alcohol.

[0078] A resin R7 is TD methyl resin, a silicone solid content is 100%, and viscosity is 200–

700csk. a resin R8 — TD methyl resin — it is — a silicone solid content — 100% — it is — T/D ratio = — it is about 49:1 15-S-9 (registered trademark) is Union Carbide (Union Carbide Corporation). It is the ethoxyl-ized secondary-alcohol system surfactant by which shell mark ting is carried out.

[0079] MINFOMU-1chi (registered trademark) is Union Carbide (Union Carbide Corporation). It is the blend of the surfactant containing the ethoxyl-ized secondary alcohol by which shell marketing is carried out. Although one of the advantages of using the micro emulsion prepared by the method of this invention is that ASTM \*\*\*\*\* can prepare the transparent personal care product of about 100-150, as shown in the example 42, into the personal care product compound of this invention, you may blend an opaque agent or a pearl foil pigment by request. If the micro emulsion prepared by the method of this invention is used, the useful conditioning effect will be given to various personal care products including the constituent for hair coloring, a rinse, the lotion for neutralization, a cream, gel, a mousse, aerosol, and a pump spray (however, not restricted to these).

[0080] By the constituent and method of this invention which were explained above, many of other deformation and corrections can be made, without deviating from the method and constituent of this invention substantially so that clearly from the above thing. Therefore, the embodiment of said this invention is mere instantiation, and any meanings do not plan to restrict a claim.

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[Translation done.]